REMARKS

These amendment and remarks are filed in response to the final rejection mailed July 3, 2007. For the following reasons, this application should be allowed and the application passed to issue. No new matter is introduced by this amendment. The amendments to claims 1, 2, and 6 are supported throughout the specification, including page 7, lines 10-12.

Claims 1-6 are pending in this application. Claims 1-6 have been rejected. Claims 1, 2, and 6 have been amended in this response.

Interview Summary

Applicants greatly appreciate the courtesy of Examiner Chu in granting a personal interview with the undersigned on September 18, 2007. During the interview, the undersigned explained differential scanning calorimetry, the endothermic amount, the relational expression in claim 2, and why the claims were enabled and definite. In addition, the undersigned asserted that the claimed invention provides unexpected results and are thus allowable over the cited references. The Examiner indicated that further consideration of the arguments would be required.

Objection to the Specification

The specification is objected to because the equation $90 < Y + 50.5X < 100$ is allegedly not understood. The Office Action pointed out that when Y and X are at the highest values the disclosed relationship is not met. This objection is traversed, and reconsideration and withdrawal thereof respectfully requested.

The Office Action mistakenly asserted that the highest value of Y is 1.0 J/g. However, 1.0 J/g is the endothermic amount for only the range of 20 to 45 °C, **not** the range of 20 to 100 °C. This relationship is clearly described by the equation and would have been readily understood by one of skill in this art in light of the teaching in the specification.

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The equation will be solved for Examples 2 and 3 from Table 1 of the present specification to show that it is understandable.

Example 2: $90 < 12.2 + 50.5(1.65) < 100$

$90 < 95.525 < 100$

Example 3: $90 < 11.2 + 50.5(1.65) < 100$

$90 < 94.525 < 100$

To facilitate understanding of the present invention, further explanation of the disclosure is provided. The entire endothermic amount referred to in Table 1 is the endothermic amount of the positive electrode current collector of the positive electrode current collector in the range of 20 to 100 °C. The endothermic amount of the positive electrode current collector in this temperature range is solely due to the endothermic amount of the wax component. The endothermic amount (J/g) listed in Table 1 is the endothermic amount of the wax per 1 g of the positive electrode current collector.

The difference between the endothermic amount in Example 1 and Comparative Example 1 is based on the difference in the wax content in the positive electrode current collector, which is based on the difference in the carbon rod density.

The endothermic amount at 45 °C or below in Table 1 is the wax endothermic amount in the temperature range of 20 to 45 °C in the entire endothermic amount of wax in the range of 20 to 100 °C.

Claim Rejections Under 35 U.S.C. § 112

Claims 1-6 were rejected under 35 U.S.C. § 112, first paragraph, because the specification is allegedly not enabled for the paraffin wax over the entire claimed range of molecular weight and carbon rod densities. The Office Action averred that the claimed invention

would cause undue experimentation by one of ordinary skill in this art. This rejection is traversed, and reconsideration and withdrawal thereof respectfully requested.

One of ordinary skill in this art would have recognized that the full scope of the claims is enabled. Measurements of density, and endothermic properties via DSC are well known and are common measurement and analysis techniques that are well within the abilities of one of skill in the art. For the Examiner's convenience a printout about the Diamond™ Differential Scanning Calorimeter from the Perkin-Elmer website is attached. The printout establishes that DSC is a well-known, well-established analytical technique. Measurements of density, and measurements of endothermic properties via DSC are well-known and common measurement and analysis techniques that are well within the abilities of one of skill in this art. The endothermic amount is the amount of thermal energy (Joules) absorbed per gram of sample within the specified temperature range. Though the Office Action alleged that the maximum endothermic amount is 1.0 J/g, Table 1 clearly teaches endothermic amounts of 10.2 to 12.2 J/g.

The Office Action has not fulfilled the requirements of asserting that the invention is not enabled. The PTO has the burden of establishing that the invention is not enabled. However, the PTO has improperly shifted the burden to Applicants to prove that the invention is enabled. The data in Table 1, shows that positive electrode current collectors that meet the claim limitations have an unexpected improvement in discharge performance. The Office Action appeared to assert that the properties of the positive current collectors are unpredictable. However, such unpredictability, rather than supporting the Office Action's assertion of lack of enablement, actually supports Applicants' position that the claimed invention is not obvious.

Claims 2 and 3 were rejected under 35 U.S.C. § 112, first paragraph, as failing to comply with the enablement requirement because Y and X have different units and, therefore, are not

combinable. This rejection is traversed, and reconsideration and withdrawal thereof respectfully requested.

First, there is no requirement that the units in a claimed relationship must be the same. Second, Applicants can be their own lexicographer and define claim limitations in any manner that would be understood by one of skill in this art. The claimed relational expression is clear and definite to one of skill in the art, and the units for Y and X are defined. Contrary to the Office Action's assertion, the numerical values of X and Y can be summed and as long as the sum of Y and 50.5X fall between 90 and 100, the limitation is met. As shown above, the relational expression has been solved for several examples. Further, Applicants have discovered that positive electrode current collectors for a manganese dry battery that meet the claimed relational expression and the other limitations provide unexpectedly improved discharge performance and voltage drop, as explained in the present specification.

Claims 2 and 3 were rejected under 35 U.S.C. § 112, first paragraph, as failing to comply with the enablement requirement because when Y and X are at the highest values the claimed relationship is allegedly not met. This rejection is traversed, and reconsideration and withdrawal thereof respectfully requested.

The Office Action mistakenly asserted that the highest value of Y is 1.0 J/g. However, as is clear from the express claim language, 1.0 J/g is the endothermic amount of the paraffin wax or microcrystalline wax per 1 g of the positive electrode current collector for only **the range of 20 to 45 °C**, not the range of 20 to 100 °C. The relationship described by the equation would be readily understood by one of skill in this art in light of the claim considered as a whole. As previously explained, 1.0 J/g is not the highest value of the endothermic amount, and examples

have been provided showing that the relational expression can be solved and is satisfied by the claimed positive electrode current collectors.

Claims 1-6 were rejected under 35 U.S.C. § 112, second paragraph, as being indefinite because the limitation "endothermic amount" is not understood. This rejection is traversed, and reconsideration and withdrawal thereof respectfully requested.

As explained above, differential scanning calorimetry (DSC) is an established analytical technique and one of ordinary skill in the art would have readily recognized that "endothermic amount" is the amount of thermal energy (Joules) absorbed per 1 g of sample as the sample temperature is raised within the specified temperature range.

The Office Action additionally questioned the significance of 1.0 J/g. The present inventors have discovered that positive electrode current collectors with an endothermic amount of more than 1.0 J/g obtained by DSC at 20 to 45 °C tend to elute more wax from the carbon rod at 45 °C inducing the sealing agent to melt, as explained in the specification in the paragraph bridging pages 4 and 5. 45 °C is significant because it is the temperature at which the manganese dry batteries are stored to evaluate high temperature storage.

Applicants submit that the claims fully comport with the requirements of 35 U.S.C. § 112.

Claim Rejections Under 35 U.S.C. § 103

Claims 1 and 4-6 were rejected under 35 U.S.C. § 103(a) as being unpatentable over Nobuaki (JP 3-297063) in view of Nagasawa et al. (U.S. Pat. No. 4,157,317). This rejection is traversed, and reconsideration and withdrawal thereof respectfully requested.

The Office Action asserted that Nobuaki discloses impregnating a carbon rod in a manganese dry cell with a hydrocarbon having a molecular weight of 300 to 500. The Office

Action acknowledged that Nobuaki does not teach the density of the carbon rod. The Office Action alleged that Nagasawa et al. disclose that a carbon rod having a density within the claimed range provides sufficient strength and allows gases to escape, thus preventing cracking. The Office Action considered the claimed endothermic amounts to be an intrinsic property of the paraffin wax having a molecular weight of 300 to 500. The Office Action further advised Applicants to prove all of the different combinations of the prior art carbon rod density and waxes would not provide an endothermic amount of less than 1.0 J/g.

Nobuaki and Nagasawa et al., whether taken alone or in combination, do not suggest the claimed positive electrode current collector. The PTO's apparent attempt to place the burden of **disproving all the prior art examples** on Applicants is improper. If the PTO maintains this position, Applicants request the PTO point out on what basis such a requirement can be made. Table 1 of the present specification proves that waxes having the claimed molecular weight and carbon rods with the claimed density do not inherently produce positive electrode current collectors with the claimed endothermic amount.

It is not necessary, when rebutting an obviousness rejection, to prove that all the prior art examples do not possess the claimed property. Rather, the PTO has the burden of establishing a prima facie case of obviousness, and if the Office Action does so, then Applicants have the burden of rebutting the conclusion of obviousness. Applicants do not have the burden of proving all the prior art examples do not possess the claimed property. The citation of *In re Best* in the Office Action does not support the Office Action's assertion that Applicants can be required to disprove all the prior art examples. There is no suggestion in the teachings of *In re Best* that Applicants can be required to disprove all prior art examples. As shown in Table 1, the

endothermic amount is not an inherent property, and the data in the present specification effectively rebuts the Office Action's assertion of prima facie obviousness.

During examination, claims are to be considered as a whole, and when all the limitations of the present claims are considered as a whole, the claimed positive electrode current collector would not have been obvious. In particular, the cited references do not suggest the unexpected improvement in discharge performance provided by positive electrode current collectors of the present invention, as illustrated in Table 1. As amended, the present claims exclude the high density carbon rod of Example 1. The claimed invention provides the unexpected results of excellent sealing performance and discharge performance of the batteries comprising positive electrode current collectors using low density carbon rods.

Obviousness can be established only by combining or modifying the teachings of the prior art to produce the claimed invention where there is some teaching, suggestion, or motivation to do so found either in the references themselves or in the knowledge generally available to one of ordinary skill in the art. *In re Kotzab*, 217 F.3d 1365, 1370 55 USPQ2d 1313, 1317 (Fed. Cir. 2000); *In re Jones*, 958 F.2d 347, 21 USPQ2d 1941 (Fed. Cir. 1992); *In re Fine*, F.2d 1071, 5 USPQ2d 1596 (Fed. Cir. 1988). There is no suggestion in Nobuaki and Nagasawa et al. to substitute a wax wherein an endothermic amount of the paraffin wax or the microcrystalline wax per 1 g of said positive electrode current collector obtained by differential scanning calorimetry at 20 to 45°C is not more than 1.0 J/g, and a carbon rod having a density of 1.55 to 1.65 g/cm^3 into the current collector of Nobuaki, as required by claims 1, 2, and 6, nor does common sense dictate the Office Action-asserted modification. The PTO has not provided any evidence that there would be any obvious benefit in making the asserted modification of

Nobuaki et al. *See KSR Int'l Co. v. Teleflex, Inc.*, 500 U.S. ____ (No. 04-1350, April 30, 2007) at 20.

The only teaching of a positive current collector with the claimed wax and carbon rod density is found in Applicants' disclosure. However, the teaching or suggestion to make a claimed combination and the reasonable expectation of success must both be found in the prior art, and not based on applicant's disclosure. *In re Vaeck*, 947 F.2d 488, 20 USPQ2d 1438 (Fed. Cir. 1991). The PTO has apparently relied on improper hindsight reasoning in reaching the conclusion of obviousness.

The dependent claims are allowable for at least the same reasons as the respective independent claims from which they depend, and further distinguish the claimed positive electrode current collector.

In view of the above remarks, Applicants submit that this case should be allowed and passed to issue. If there are any questions regarding this response or the application in general, a telephone call to the undersigned would be appreciated to expedite the prosecution of the application.

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To the extent necessary, a petition for an extension of time under 37 C.F.R. § 1.136 is hereby made. Please charge any shortage in fees due in connection with the filing of this paper, including extension of time fees, to Deposit Account 500417 and please credit any excess fees to such deposit account.

Respectfully submitted,

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Please recognize our Customer No. 20277 as our correspondence address.

Materials Characterization

- Differential Scanning Calorimetry -

Differential Scanning Calorimetry (DSC)

Glass Transition Temperature

Melting Point Determination

Thermomechanical Analysis (TMA)

Thermogravimetric Analysis (TGA)

Dynamic Mechanical Analysis

Microidentation

Contact Resistance Measurements

Fourier Transform Infrared Spectroscopy (FTIR)

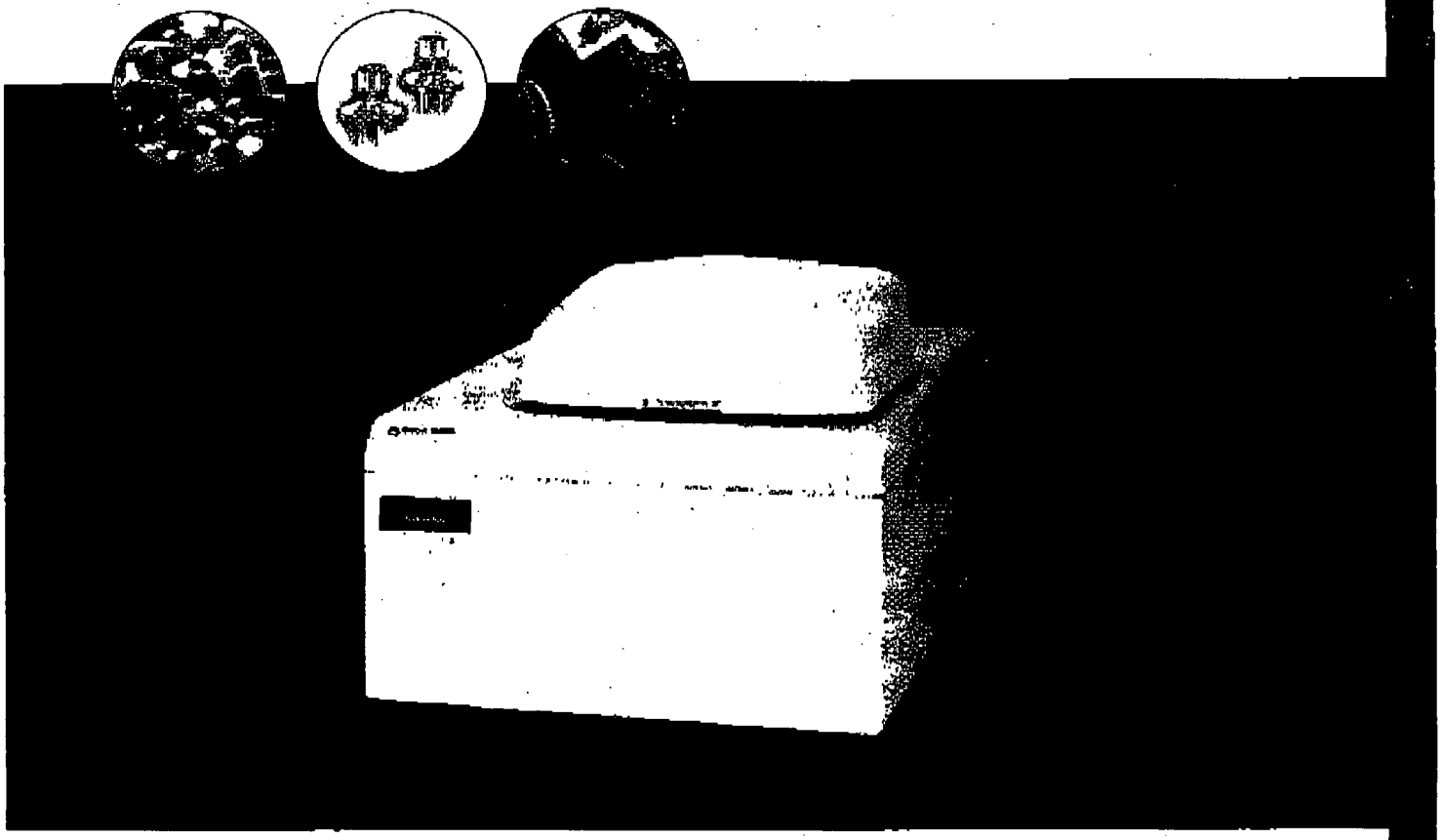
Energy Dispersive Spectroscopy (EDS)

High Performance Liquid Chromatography (HPLC)

The Perkin-Elmer Pyris-1 Differential Scanning Calorimeter (DSC) measures the amount of energy (heat) absorbed or released by a sample as it is heated, cooled, or held at a constant temperature. Typical applications include determination of melting point temperature and the heat of melting; measurement of the glass transition temperature; curing and crystallization studies; and identification of phase transformations.

The temperature range of the DSC Pyris-1 is -170°C to 730°C.

Diamond Differential Scanning Calorimeter (DSC)



high sensitivity thermal analysis

PerkinElmer®
precisely.

unique capabilities ensure clear identification

When you can't afford to miss something in your DSC analyses, you need the Diamond DSC (Differential Scanning Calorimeter). Missing important information can make a big difference to your organization's success. It can lead to product failures, additional manufacturing expense and wasted time. With the PerkinElmer-exclusive power-compensation technique, you will be confident to achieve fast, accurate, reproducible results.

PerkinElmer, the leader in high sensitivity thermal analysis instrumentation, pioneered the proven power-compensation approach to DSC nearly 40 years ago. Two small, low-mass furnaces heat and cool rapidly, providing better resolution and higher sensitivity, enabling detection of transitions that are missed in conventional DSC systems. And, since the design measures heat flow (energy), you get direct results instead of having to derive them from a temperature difference (delta T) calculation as in other DSC's.

What does DSC measure?

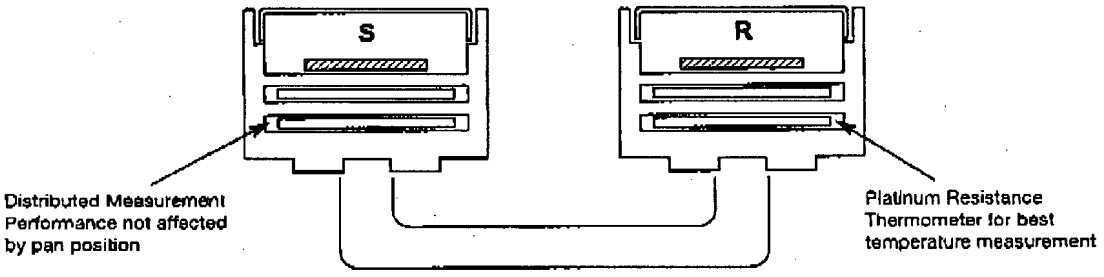
DSC measures the amount of energy (heat) absorbed or released by a sample as it is heated, cooled or held at constant temperature. DSC also performs precise temperature measurements.

QUICK GLANCE

- HyperDSC™ for unmatched sensitivity
- Low-mass furnaces for reduced analysis time and high throughput
- Superior signal resolution so you don't miss transitions
- Closed-loop operation for true isothermal measurements
- Unsurpassed calorimetry for accurate specific heat analysis

The power-compensation principle

With power-compensation DSC, the sample and the reference material are placed in independent furnaces. When the temperature rises or falls in the sample material, power (energy) is applied to or removed from the calorimeter to compensate for the sample energy. As a result, the system is maintained at a "thermal null" state at all times. The amount of power required to maintain system equilibrium is directly proportional to the energy changes occurring in the sample. No complex heat-flux equations are necessary with a power-compensation DSC because the system directly measures energy flow to and from the sample.



HyperDSC - a breakthrough method

The PerkinElmer-exclusive HyperDSC method delivers unparalleled sensitivity and new insights into materials processes that cannot be obtained with existing DSC methods. By providing sample information within seconds, HyperDSC significantly increases throughput in the polymer and pharmaceutical industries.

The HyperDSC method is only possible with the power-compensation Diamond DSC because it allows measurements with controlled scanning rates from 0.01 °C to 500 °C/minute. Unlike other DSC methods, HyperDSC offers true materials analysis while either eliminating or reducing changes such as re-crystallization, melting, and decomposition, which may be induced when utilizing slow scanning.

Fastest heating and cooling rates

Unlike conventional DSC systems utilizing large furnaces that are 30 grams or more, the Diamond DSC system features two small 1-gram furnaces. This design enables the system to achieve the fastest heating and cooling rates in the industry – up to 500 °C/min. Faster heating and cooling rates translate into higher sensitivity and throughput. Small furnaces also deliver better temperature control, providing linearity for better results.

Heating and cooling flexibility allows the Diamond DSC to more accurately mimic production conditions, facilitating process and property optimization, saving time and reducing production problems.

Better resolution means you see more

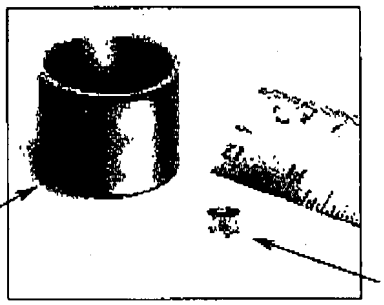
Resolution refers to the ability of the DSC to separate or resolve closely occurring thermal transitions. In thermal analysis, small differences can have a huge impact on your success. Ensuring access to all the information is critical to researchers in both the pharmaceutical and polymer industries. In pharmaceutical labs, researchers need to ensure that there are no unknown transitions, which could not only cause undesirable outcomes, but may potentially limit patent protection. In polymer work, a small shoulder peak that goes undetected can produce unwanted physical properties, resulting in product failure. Due to the low-mass furnace design of the Diamond DSC, the signal returns to baseline faster, providing outstanding resolution. The Diamond DSC delivers industry-leading performance to ensure all transitions are identified.

Higher sensitivity

Power-compensation yields higher sensitivity, which is the ability of a DSC to detect a weak transition from the background noise. You can even detect phase transitions of low energy, such as in biological or liquid crystalline samples. The Diamond DSC has the capability to analyze small amounts of material if sample availability is limited. Because the signal is directly proportional to the heating rate, the additional capability of fast scanning (HyperDSC) increases sensitivity.

Size matters

Heat-flux systems use one large furnace that is between 30 and 200 grams while the Diamond DSC system features two 1-gram furnaces. This size difference results in faster heating and cooling rates and allows direct heat flow determination, eliminating the need for complex mathematics.



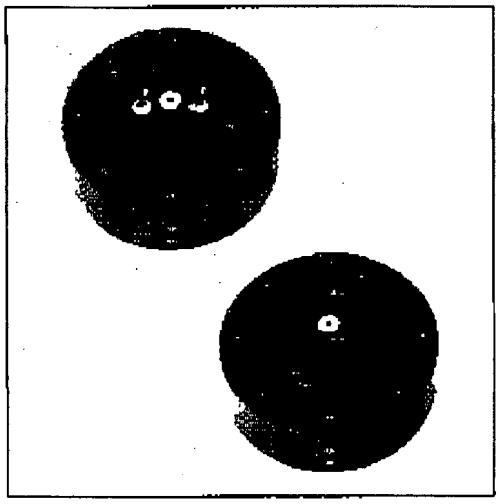
unique capabilities ensure clear identification (continued)

True isothermal measurements

Power-compensation DSC improves analysis of isothermal crystallization processes and curing reactions. By comparison, a conventional DSC performs these measurements only with limitations, since the temperature is not kept constant. During crystallization, the temperature can easily change, which influences the data obtained and results of any kinetic studies. The Diamond DSC maintains the temperature difference at zero and thereby ensures high quality results.

Cover design maximizes system baseline stability

The Diamond DSC system includes a unique cover design that improves both ease-of-use and performance. An innovative rotating sample head cover effortlessly slides in and out of position. The cover is in constant contact with the furnace block, reducing equilibrium times and ensuring baseline stability.



Rotating sample head cover.

High calorimetric accuracy

The power-compensation principle determines the most accurate calorimetric data of all available DSC's. The PerkinElmer power-compensation DSC is the benchmark for direct quantitative measurement of specific heat in a wide variety of materials. With a conventional DSC, the results vary with the sample preparation and instrument operating conditions such as contact with the sensor.

With the Diamond DSC you minimize the risk of wrong or inaccurate determination – the influence of inconsistent sample preparation on the results is negligible. You know the generated data is correct and feel secure using it as a basis for your decisions.

High pressure DSC

Designed for convenient operation with a standard DSC, the optional DSC High Pressure Cell extends the capabilities of the unique power-compensation Diamond DSC design to elevated pressure measurements. While most DSC experiments are performed at atmospheric pressure, there are some applications that must be carried out at elevated pressure and the DSC High Pressure Cell allows for such applications.

Typical applications include:

- Oxidation testing of oils, fats, foods and plastics
- Curing and crosslinking reactions
- Suppression of volatiles vaporization
- Analysis of pressure dependent chemical reactions

accessories provide limitless flexibility

The Diamond DSC accommodates a wide range of accessories including an Autosampler, automatic gas control and switching accessories, several cooling devices and the widest variety of sample pans.

Autosampler

The Diamond DSC Autosampler allows automated DSC testing of multiple samples without operator intervention. Whether running many samples using "traditional" scanning rates, or very fast HyperDSC methods on many different samples, the Diamond DSC Autosampler finds use in a wide range of laboratory settings.

The Diamond DSC Autosampler uses a pneumatic sample arm with refined electronics for precise position control. A unique carousel holds multiple sample capsules and is designed to allow convenient addition and removal of sample materials before, during or after measurement. An integral enclosure isolates the Autosampler from mechanical interference and contaminants. This third-generation Autosampler design, used in laboratories throughout the world, provides the reliability you expect when performing unattended operation.

Convenience and ease-of-use are also important considerations when automating thermal analysis experiments. The Diamond DSC Autosampler utilizes the benchmark Pyris™ software for experiment set up, control of sample exchanges, and programmed data analysis at the conclusion of an experiment. The "PlayList", a standard feature of Pyris software, is used to operate the Diamond DSC Autosampler. With this software, a list of samples can be automatically loaded/unloaded, run and the results analyzed and printed. Complete sample lists and their methods can be saved and conveniently recalled and modified later for re-use. New samples or emergency "rush" samples can be added at any time, offering the ultimate in flexibility.

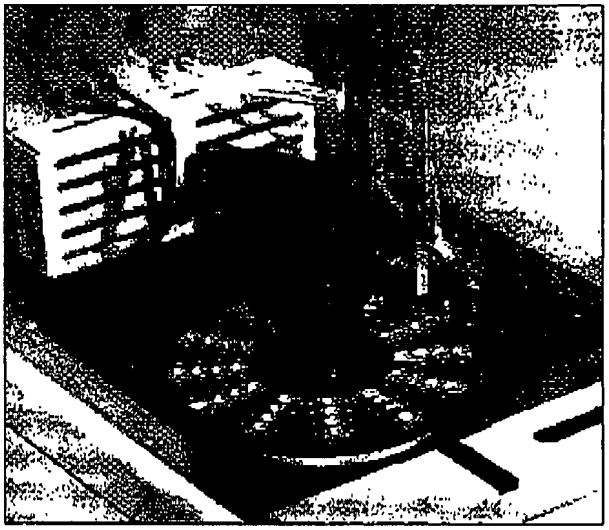
The Diamond DSC Autosampler and Pyris software do more than increase sample throughput and run your Diamond DSC continuously. They make running thermal analysis faster and easier for operators at all levels of experience, while providing accurate and reproducible results.

Enhanced cooling capabilities

A variety of cooling options are available for the Diamond DSC, including liquid circulation, a refrigerated cooling system and an automated liquid nitrogen cooling accessory, allowing experiments that require subambient analysis temperatures or fast controlled cooling steps.

Sample pans

A wide assortment of sample pans are available from PerkinElmer to address the range of thermal analysis applications. You can choose from different pan materials (aluminum, gold, platinum, graphite, aluminum oxide, copper, silver, stainless steel), capacities (a few µL to 50µL) and pressure ratings (ambient to 150 atmosphere) to optimize your analysis.



44-position Diamond DSC Autosampler.

6

Pyris software improves lab productivity

Pyris software is the benchmark application for thermal analysis. In combination with the Microsoft Windows® operating system, it provides new functionality essential for productive operation of all thermal analysis techniques. Whether in a totally automated research laboratory, an automated QA/QC lab, or a stand-alone instrument, you can be sure that the proven Pyris software meets your operating requirements.

Report Manager

The Pyris Report Manager gives you the capability of exporting a Pyris data file to a document in Microsoft® Word or HTML (Hypertext Markup Language) format. The software provides user control of the report and of the content of the report. This report template can be re-used to conveniently facilitate the creation of new reports.

Calibration Wizard

The integrated Calibration Wizard offers two levels of calibration control. E-Z Cal, based on predefined conditions, allows fast calibration with minimal user interaction. If greater flexibility or accuracy is required, Advanced Cal provides the ability to calibrate using multiple temperature and heat flow standards, enabling the calibration to be customized for the temperature range of interest. Advanced Cal provides an algorithm which allows the use of a certified Sapphire standard in accordance with ASTM to calibrate heat flow. In addition to offering two levels of control, the Calibration Wizard utilizes Autotune for automatic baseline optimization.

Valet

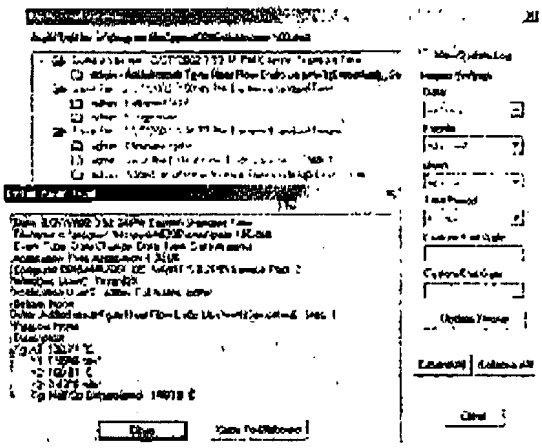
The convenient Valet feature allows creation of pre-set start-up and shut-down events. For example, valet can be used to automatically condition and equilibrate the Diamond DSC prior to running experiments, or it can be used to automatically shut down the system after completion of runs.

Pyris Enhanced Security for regulatory compliance

Pyris Enhanced Security, an add-on to Pyris software, helps users in both research and quality control to comply with stringent data security requirements, including the 21 CFR Part 11 mandates of the U.S. Food and Drug Administration.

With Pyris Enhanced Security, users in regulated industries will be confident in their ability to provide the whole story about the generation of data from their thermal analyzers. It provides all of the required 21 CFR Part 11 technical compliance features to ensure that data integrity is always maintained:

- User Level Management & Security
- File Protection
- Audit Trails
- Electronic Signatures



Audit Trail records all significant events pertaining to data and system changes which are displayed in a Viewer.

StepScan DSC

StepScan DSC is a modulated temperature DSC technique that operates in conjunction with power-compensation DSC. The approach applies a series of short interval-heating and isothermal-hold steps to cover the temperature range of interest. This approach requires a DSC with very fast responsiveness to achieve short-interval linear heating and isothermal steps. The use of the ultra-low mass furnaces (1 g) with the power-compensation DSC ensures the fastest response time of any DSC instrument. StepScan DSC offers many advantages over conventional DSC and other MTDSC heat-flux DSC approaches:

- Straightforward — pure linear-heating ramps and isothermal steps
- No sine waves and possibilities of distortions and other experimental artifacts
- No mathematical deconvolution (Fourier transforms) required with associated complexities
- Highly quantitative
- Eliminates need to perform heat-cool-reheat experiments
- Greatly helps in data interpretation since reversible and irreversible effects are separated out
- Provides clearer identification of the glass transition event (Tg)
- Yields more accurate heat-capacity results since Cp measurements are generated over short-interval temperature segments

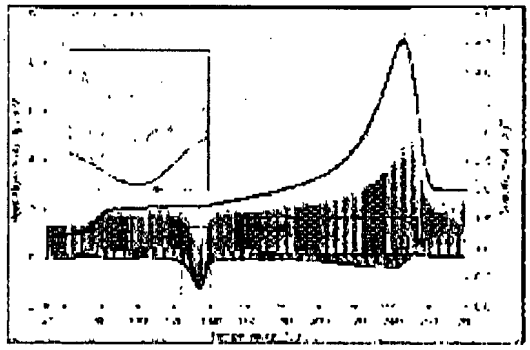
StepScan: A Pure Approach to MTDSC Applications

With the StepScan DSC approach, two signals are obtained. Thermodynamic Cp signal represents the reversible aspects of the material, while the Iso K signal reflects the irreversible nature of the sample during heating. The following basic equation mathematically describes the StepScan DSC approach:

Heat Flow = Cp(dT/dt) + f(T,t)

In this equation, Cp is the sample's heat capacity, dT/dt is the applied heating rate and f(T,t) is the kinetic response. The first Cp term represents the reversible aspects of the sample and the power-compensation DSC applies a purely linear heating ramp for the best results rather than a sine wave where the first item is continuously varying. When the sample is held under isothermal conditions, the heating rate becomes 0 and the sample's heat flow is purely described by the kinetic term.

Because the sample is either linearly heated or held isothermally (true isothermal), the StepScan DSC approach is straightforward and provides the purest and fastest approach to MTDSC measurements.



StepScan DSC Results for PET.

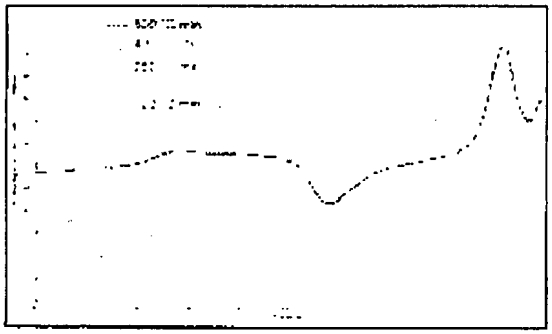
HyperDSC significantly increases sensitivity and throughput

HyperDSC in pharmaceuticals

HyperDSC simplifies identification of the glass transition in amorphous materials

There has been great interest over the years in the study of the Glass Transition (Tg) of amorphous lactose. Lactose is a very important excipient for pharmaceuticals and is used widely as a diluent in the formulation of tablets. A spray-dried lactose that approached 100% amorphous content as determined by solution calorimetry was analyzed with the Diamond DSC. Data were collected with conventional scanning rates of 20 and 100 °C/min, and the HyperDSC data are collected at the scanning rates of 250, 400, and 500 °C/min. All the scans were performed on the same analyzer. The Tg of lactose is normally seen in the temperature range of 100-120 °C and is difficult to identify using conventional DSC scanning rates. HyperDSC increases the sensitivity and shows the transition clearly. The Tg found with HyperDSC was in the temperature range of 80-100 °C. This lower Tg temperature is believed to be caused by the plastization of the lactose by water, which is not lost during the fast scan. After the glass transition, an exotherm associated with re-crystallization is observed, and this is followed by two melting events. The two peaks are associated with the two forms of lactose that re-crystallize from the post Tg material. The first peak is the melting of anhydrous α lactose, and the second peak is associated with β lactose.

Note: Acknowledgement to Paul Gabbott, Paul Clarke, Tim Mann, Paul Royall and Sukhraj Shergill for their initiative and work in this field of application.

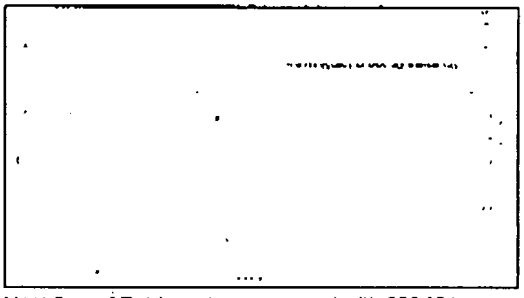


Analysis of amorphous lactose at HyperDSC and conventional DSC scanning rates.

HyperDSC in pharmaceuticals

HyperDSC as a screening tool

An unfounded concern using HyperDSC is the loss of resolution. This example shows the run of Dotriacontane with 250 °C/min and the second derivative, a very valuable tool for the use of HyperDSC — it clearly shows several transitions which are not resolved in the high scanning rate heat flow curve. The information can be generated in less than two minutes and helps in the selection of samples which may provide additional information at lower scan rates.



Heat flow of Dotriacontane, scanned with 250 °C/min.

HyperDSC in polymers

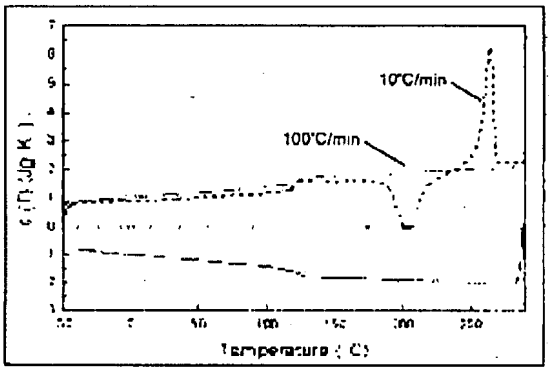
HyperDSC helps mimic process conditions

The plastics industry has a high interest in having a clear understanding of how materials behave under process conditions. In injection molding, blow-molding and extrusion processes, high cooling and heating rates play the dominant role. Product development requires lab-scale experiments in which these conditions can be simulated. HyperDSC provides that additional information that you don't capture with standard DSC. In addition, high heating and cooling rates minimize noise, thereby enabling highly quantitative measurements.

The analysis of a PET sample demonstrates the influence of the scanning rate on the results.

The slow heating at 10 °C/min after cooling the PET at 100 °C/min shows extensive cold crystallization followed by a melting of the formed crystals (dashed line). This heating run performed at 10 °C/min is definitely not representative of the thermal history (blue line) in which no crystallization event occurs. Heating at 100 °C/min (red curve) after cooling at 100 °C/min shows hardly any cold crystallization on the time scale of the experiment. With an increasing heating rate, recrystallization is suppressed and the resulting heating curve reflects the characteristic of the material at room temperature obtained by a fast cooling.

Note: Acknowledgement to Vincent Mathot, Thijs Pijpers, Eric van der Vegte, DSM Research, The Netherlands for their initiative and work in this application field.

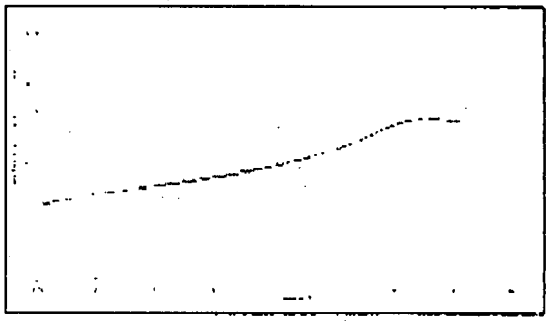


PET Analysis by HyperDSC: No cold crystallization during fast scanning.

HyperDSC in polymers

HyperDSC rapidly characterizes materials with weak transitions

Conventional DSC analysis often does not provide the high sensitivity required for the determination of weak glass transitions in highly crystalline materials or filled polymers. Techniques such as dynamic mechanical analysis (DMA) or modulated temperature DSC are often used to analyze the transition temperature in these cases. Unfortunately, both approaches are very time consuming and may require highly experienced technicians or scientists to run and analyze the data. HyperDSC provides a way to use DSC analysis for these applications, simplifying the experimental method and analysis of data. This example shows the measurement of an epoxy which is highly filled with glass fiber analyzed with a HyperDSC method (scanning rate of 250 °C/min). The increased sensitivity with HyperDSC provides an unambiguous glass transition event in less than one minute as shown here. To demonstrate the reproducibility of the Diamond DSC in the HyperDSC mode, a second sample was analyzed. The results show a nearly identical curve for both sample runs.



Glass transition in a highly-filled epoxy analyzed by HyperDSC.

10

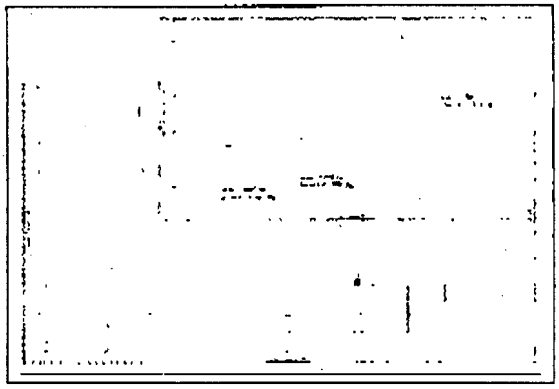
power-compensation technique yields confidence-building results

High sensitivity

Effective competitive analysis requires high resolution and sensitivity

Liquid crystalline materials can exhibit a wide range of liquid crystalline phases with varying degrees of molecular order. These changes can be seen by DSC studies but the energies involved in these transitions can be very small, making their identification difficult. In the past, most of these small energy transitions have been identified by optical microscopy alone. In this example, we show that it is possible to see these small energy transitions plainly with the Diamond DSC.

If the DSC trace is zoomed into the area of 80-95 °C, it is possible to identify some small endothermic events that occur in this range. The energies involved in these transitions are very small (0.024J/g) and demonstrate the extremely high sensitivity of the Diamond DSC.



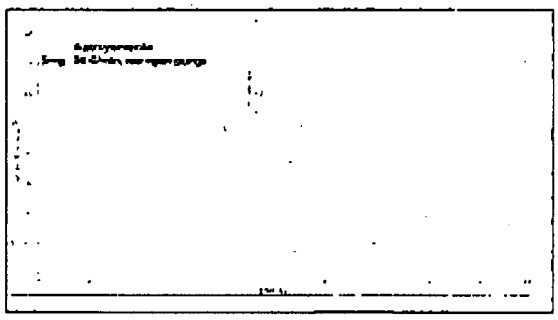
Liquid crystal phase change transitions.

Proof of performance

Oftentimes, instrument companies provide specifications as to sensitivity and resolution without describing how they were generated

The use of 4, 4' azoxyanisole has been recommended as both a resolution and sensitivity standard for assessing DSC performance by the Netherlands Society for Thermal Analysis (TAWN). This substance has two closely occurring endothermic transitions at 117 °C and 134 °C. The resolution of the DSC instrument can be defined as how well the heat flow response returns to a linear baseline in the region between the two transitions when the sample (5 mg) is heated at a rate of 20 °C/min using a nitrogen purge gas. The better the resolution of the instrument, the better the heat flow response returns to the baseline. An instrument with a high inherent resolution such as the Diamond DSC will yield a linear heat flow response between the two transitions and a better return to the baseline.

The resolution index, or R value, can be used to compare DSC performance. This is done by taking the heat flow value at the minimum point between the two peaks and then dividing this by the value of the heat flow at the smaller peak maximum.



DSC results on azoxyanisole test.

Diamond DSC
specifications

DSC Type	Power-compensation temperature null principle. Measures temperature and energy directly, rather than differential temperature (DT).	
DSC Cell	Independent dual furnaces constructed of platinum-iridium alloy with independent platinum resistance heaters and temperature sensors with furnace mass less than 1g.	
Temperature Sensors	Distributed, Platinum Resistance Thermometers for best linearity.	
Atmosphere	Static or dynamic, including nitrogen, argon, helium, carbon dioxide, air, oxygen or other inert or active gases, over full temperature range. Oxygen can be used up to 730 °C which allows easy cleaning.	
Temperature	Range Accuracy / Precision	-170 °C to 730 °C ±0.1 °C / ±0.01 °C
Calorimetry	Accuracy / Precision Sensitivity Dynamic Range	< ±1% / < ±0.1% 0.2 µW 0.2 µW to 800 mW
Signal Response	(1 mg Indium, 10 °C/min, nitrogen purge) Peak Height Width at half height H/W Ratio	 7.44 mW ± 0.15 mW 0.42 ± 0.10 °C 17.6 mW/°C ± 1 mW/°C
Isothermal Drift (10 min)	-150 °C / 100 °C	< 15 µW / < 10 µW
Scanning Rates	Heating/Cooling	0.01 °C to 500 °C/min
Temperature Overshoot	100 °C/min	< 0.1 °C
Controlled Cooling	Ambient Coolant - nitrogen purge Liquid N_2 Coolant - helium purge 	 10 °C/min to 50 °C 20 °C/min to 65 °C 50 °C/min to 100 °C 100 °C/min to 170 °C 10 °C/min to -170 °C 50 °C/min to -165 °C 100 °C/min to -135 °C 200 °C/min to -85 °C 300 °C/min to -80 °C 400 °C/min to -10 °C
Cooling Times	Ambient Coolant Liquid N_2 Coolant	725 °C to 100 °C (under 4 minutes) 200 °C to -150 °C (under 2 minutes)
Cooling Options	Ice Water Circulating Liquid Refrigerator (Intracooler) Automatic Liquid N_2 (CryoFill)	25 °C to 730 °C -10 °C to 730 °C -70 °C to 730 °C -170 °C to 300 °C
Autosampler	The Diamond DSC Autosampler can run up to 44 sample positions unattended. It has the ability to be customized through Pyris Player to meet your analysis needs and increase productivity.	
High Pressure Cell	Extends the capabilities of the power-compensation Diamond DSC design to elevated pressure measurements. Pressure range is up to 42 bar (600 psi).	
Quality Assurance	Developed under ISO 9001	
Dimensions (HxWxD)	34 x 40 x 67 cm (14 x 16 x 27 in)	
Weight	20 kg (44 lbs)	
Power Requirements	100-240 Volt, 50/60 Hz	

thermal analysis solutions

for material property analysis

PerkinElmer is the leader in high sensitivity thermal analysis instrumentation, providing you the confidence to achieve fast, accurate, reproducible results.

Differential Scanning Calorimetry (DSC)

DSC measures the amount of energy absorbed or released by a sample as it is heated, cooled or held at a constant temperature. This technique is used for polymer and pharmaceutical applications. PerkinElmer offers the best of both worlds – the Diamond DSC for highest resolution and sensitivity, and the Sapphire DSC and Pyris 6 DSC for ease-of-use and robustness.

Thermogravimetric Analysis (TGA)

TGA measures the change in weight of a sample as it is heated, cooled or held at a constant temperature. The PerkinElmer Pyris TGA instruments provide robustness and reliability for quality control and the answers researchers need to solve even the toughest problems.

Thermogravimetric/Differential Thermal Analysis (TG/DTA)

TG/DTA is a simultaneous technique that determines the weight change of a sample (TG) and measures the change in temperature between a sample and the reference as a function of temperature and/or time (DTA). The Diamond TG/DTA combines the high flexibility of the differential analysis feature (DTA, DSC) with the proven capabilities of the thermogravimetric (TG) measurement technology to provide highly reliable characterization information.

Dynamic Mechanical Analysis (DMA)

DMA measures changes in mechanical behavior, such as modulus and dampening, as a function of temperature, time, frequency, stress or a combination of these parameters. The Diamond DMA, with its more than 20 patents, provides state-of-the-art measurement to a wide variety of materials and applications.

Thermomechanical Analysis (TMA)

TMA determines dimensional changes in materials as a function of temperature or time. It is used to measure changes in length, width, thickness and linear expansion of materials. The Diamond TMA is an advanced thermomechanical analyzer which allows for samples to be analyzed using dynamic force, providing basic DMA data besides many TMA features, continuing the tradition of high sensitivity TMA.

OneSource℠ Laboratory Services — comprehensive service and support for today's results-driven lab

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